

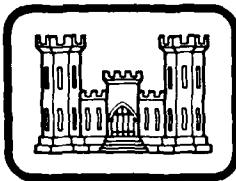
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EXAMINATIONS OF SIMULATED BOREHOLE SPECIMENS

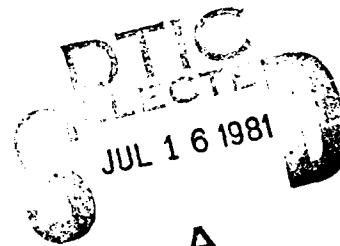
by

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May 1981
Final Report

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Prepared for Office of Nuclear Waste Isolation
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The quality of the contact between portland cement grout and surrounding rock will be of prime importance if grout is used to seal boreholes for the isolation of nuclear waste. The contact will need to be tight and relatively impermeable.	In the study reported herein, simulated borehole (SBH) specimens were prepared and tested in the laboratory. The specimens consisted of grout (Continued)	

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20. ABSTRACT (Continued)

poured into a hole in sections of anhydrite rock core that had been grouted into a steel pipe to provide restraint. The study was largely devoted to investigating methods of avoiding artifacts during preparation of these SBH specimens for study of the grout-to-rock contact. The work was conducted and is reported in two parts. The first part was a study in which only large round SBH specimens (about 6 by 6 in.) were used, while the second also included some smaller round SBH specimens (about 3 by 3 in.).

The following were among the findings:

- a. The type of specimen (SBH) merits study as a simulation of the grout-to-rock contact in a borehole or other cavity.
- b. Since the ends of the SBH specimens are invariably smeared over with grout, fresh fracture surfaces from these specimens are needed for such studies.
- c. The restraining ring should be kept in place to avoid stress relief cracking of the SBH specimens.
- d. Drying of the SBH specimens should be avoided since this leads to cracking along the contact surface (grout-to-rock) and in the grout.
- e. Since specimen drying is a usual part of preparation before scanning electron microscope examination, this method of examination should not be used unless special precautions to avoid drying can be implemented.
- f. A method of keeping an SBH specimen continuously wet during sample preparation and examination was developed and was found satisfactory.
- g. Examination of the grout-to-rock contact in a wet SBH specimen with a stereomicroscope is recommended.

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PREFACE

The work described in this report was funded by the U. S. Department of Energy and Sandia National Laboratories. It was begun in connection with studies conducted in cooperation with Sandia. It was continued and this report was prepared under Contract No. DE-AI97-81ET46633. The work was identified as "Investigation of Composition, Constitution, Properties, and Interactions of Materials Considered for Use in Plugging Boreholes and Shafts in Connection with Nuclear Waste Isolation."

Mr. C. W. Gulick was Project Officer for the portion of the work done for Sandia. Mr. Floyd L. Burns of the Office of Nuclear Waste Isolation, Battelle Memorial Institute, Columbus, Ohio, was Project Manager for the latter part of the work.

The work was done in the Structures Laboratory of the U. S. Army Engineer Waterways Experiment Station (WES) under the general direction of Mr. Bryant Mather, Laboratory Chief, and Mr. John M. Scanlon, Jr., Chief, Concrete Technology Division. Messrs. John A. Boa, Jr., and Donald M. Walley of the Grouting Unit made the specimens and provided technical assistance. Mrs. Katharine Mather was Project Leader. This report was prepared by Messrs. Jay E. Rhoderick and Alan D. Buck.

CONTENTS

	<u>Page</u>
PREFACE	1
CONVERSION FACTORS, INCH-POUND TO METRIC (SI)	
UNITS OF MEASUREMENT	3
PART I: FIRST STUDY	4
Introduction	4
Experimental Procedure	5
Results and Discussion	8
Preliminary Conclusions	12
PART II: SECOND STUDY	14
Introduction	14
Experimental Procedure	14
Results	17
Conclusions	17

CONVERSION FACTORS, INCH-POUND TO METRIC (SI)
UNITS OF MEASUREMENT

Inch-pound units of measurement used in this report can be converted to metric (SI) units as follows:

Multiply	By	To Obtain
Fahrenheit degrees	5/9	Celsius degrees or Kelvins*
inches	25.4	millimetres
pounds (force) per square inch	6.894757	kilopascals
pounds (mass)	0.45359237	kilograms

* To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula: $C = (5/9)(F - 32)$. To obtain Kelvin (K) readings, use: $K = (5/9)(F - 32) + 273.15$.

EXAMINATIONS OF SIMULATED BOREHOLE SPECIMENS

PART I: FIRST STUDY

Introduction

1. In work concerned with plugging boreholes using grout mixtures to isolate nuclear waste repositories and in related geochemical studies, the Office of Nuclear Waste Isolation (ONWI) has found that the type and quality of contact between the grout and the host rock are important considerations.* It is important that the contact between host rock and grout be tight and of relatively low permeability.

2. Examinations of simulated borehole specimens resembling those described in this report were made and reported by the Oak Ridge National Laboratory (ORNL).** ORNL examined the specimens with a scanning electron microscope which required vacuum- or freeze-drying that is believed to have opened a crack at the contact between the central grout filling and the anhydrite annulus.

3. The Structures Laboratory, WES, examined some untested specimens which had been continuously cured immersed and then in air, and observed that a crack formed after drying had begun. Other specimens were prepared and examined later to compare the effects on specimens of drying and continual immersion. The first of these examinations in late 1979 and early 1980 used specimens cast in annuli in 4-in.-OD (outside diameter)† anhydrite cores restrained by expansive grout in 6-in.-ID steel pipe with a wall thickness of approximately 1/4 in. The later

* ONWI. 1979a, 1979b, and 1980. "Development of Plan and Approach for Borehole Plugging Field Testing," "Status of Borehole Plugging and Shaft Sealing for Geologic Isolation of Radioactive Waste," and "NWTS Repository Sealing Program Plan," Report Nos. ONWI-3, ONWI-15, and ONWI-54, Battelle Memorial Institute, Columbus, Ohio.

** Personal communication from John Moore, ORNL, to Katharine Mather, Structures Laboratory, U. S. Army Engineer Waterways Experiment Station (WES), 9 November 1979.

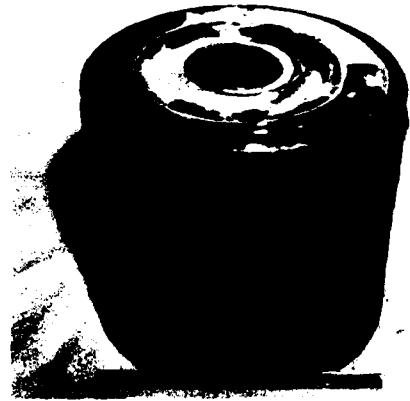
† A table of factors for converting inch-pound units of measurement to metric (SI) units is presented on page 3.

part of the examinations made in late summer 1980 used some specimens cast in 6-in.-ID or smaller steel pipe and five specimens cast in 2-3/4-in. OD steel pipe that was 3 in. long.

Experimental Procedure

4. Three specimens were prepared in the laboratory using 4-in.-OD anhydrite (anhydrous orthorhombic CaSO_4) cores as the host rock. Figure 1 shows the size and appearance of the specimens. Each was intended to

Figure 1. Specimen A10 as cast, X0.3. The uneven white masking on the top surface shows why slices were cut normal to the vertical axis to examine cleaner sawed surfaces. The central circular plug is grout, the surrounding annulus is anhydrite, the outer annulus is grout, and the exterior restraining ring is 6-in.-ID steel pipe



simulate conditions in a borehole after a grout plug had been placed to seal it. The anhydrite core was 4 in. in diameter from which a 2-in.-OD core was drilled providing an annulus with walls averaging 1 in. thick (the 2-in. holes were not always centered in the 4-in. cores). The steel pipe encasing the perimeter of the specimen was intended to provide the restraint provided in the borehole by the rock in place. The expansive grout in the central annulus represented the borehole plug; the outer annulus of expansive grout between the rock and the pipe was intended to fill the excess space and restrain the anhydrite annulus. The grout in the outer annulus was placed in the same sequence of operations as that in which the center plug was placed. The situation simulated in the first three specimens was the first Bell Canyon Test* plug placed in Hole AEC-7 near Carlsbad, N. Mex., 26 September 1979, in anhydrite host rock.

* Gulick, C. W., Boa, J. W., Jr., and Buck, A. D. 1980a and 1980b. "Bell Canyon Test (BCT) Cement Development Report," and "Bell Canyon Test (BCT) Cement Grout Development Report," Report Nos. SAND 80-0358C and SAND 80-1928, Sandia National Laboratories, Albuquerque, N. Mex.

The anhydrite cores used in the laboratory specimens came from Hole AEC-7; groundwater from Hole AEC-7 was used to cure the laboratory specimens.

5. Grout mixture BCT-1-FF, an expansive grout with fresh mixing water used in the September 1979 Bell Canyon Test plug, was also used in the specimens cast in the laboratory. Other similar specimens were used to test the permeability of the interface between the central grout plugs and the surrounding rock. All these specimens were cylindrical and about 6 in. tall and 6 in. in diameter (Figure 1). Details of casting and curing of the specimens examined are presented below:

Specimen No.	Date, 1979		Method of Placing Grout Filling	Curing
	Cast	Received		
2*	21 Aug	16 Nov	Grout poured into the central core hole and between the outside of the anhydrite and the steel pipe	Filled specimen was immersed in 10 lb of brine water at 1500 psi and 150°F (65.6°C) for 7 days. The specimen was then removed from the water, wrapped in plastic, and stored at 73.4°F (23°C) for 87 days total
A10	20 Nov	4 Dec	Grout placed as for specimen 2. Mixing water saturated with CaSO_4	Filled specimen was immersed in groundwater from Hole AEC-7 at the Waste Isolation Pilot Plant (WIPP) site for 14 days at 100°F (38°C) and 1500 psi
A7	21 Nov	30 Nov	The central core hole and the outer annulus were filled with groundwater from the WIPP site and grout poured into each brine-filled space to displace the brine	Filled specimen was immediately immersed in groundwater from Hole AEC-7 at the WIPP site for 7 days at 100°F (38°C) and 1500 psi

* Leaked during permeability test before petrographic examination.

6. During filling of the space in and around the rock with grout, the contact area at the top of the specimen was smeared so that later observation of the contact was not practical. Consequently, slices normal to the length of the cylinder were sawed dry with a diamond saw, or the specimen was fractured normal to its vertical axis. The specimens received for examination were:

- a. Specimens 2 and A7. Four slices from each specimen, each about 1/4 in. thick, were obtained. Two came from the top and two from the bottom of the specimens. The metal restraining ring had been removed from most of the slices. The contact area in the slices from specimens 2 and A7 was examined to determine depth of penetration of brine from the storage tank along the contact zone of the specimen and to determine why the grout was cracked.
- b. Specimen A10. A 1-in.-thick slice cut from the top part of the specimen was examined. The metal restraining ring was still in place.

7. The sawed surfaces of the nine slices from the three specimens were examined with a stereomicroscope. Special attention was paid to the contact of the grout plug with the surrounding rock. Several photographs were taken at less than 1X and at low magnification to show features of interest.

8. Small pieces of grout were cut dry from a slice of specimens 2 and A7. A surface of these pieces was ground in methanol and examined by X-ray diffraction in an atmosphere of static nitrogen. A beaker of hot barium hydroxide or a sponge soaked in barium hydroxide was added to help prevent carbonation and dehydration of the grout during examination.

9. White material from the contact area between the grout and rock of specimens 2 and A7 was removed by hand-picking, ground in methanol, allowed to dry on a glass slide, and examined by X-ray diffraction. All X-ray patterns were made with an X-ray diffractometer using nickel-filtered copper radiation.

10. The slice of specimen A10 was dried overnight in a vacuum desiccator at 140°F (60°C) to prepare it for examination with a scanning electron microscope.

Results and Discussion

11. Figure 1 shows the general appearance of a simulated borehole specimen. The surfaces that were examined after cutting or fracturing were all normal to the vertical axis of specimens like the one shown.

12. The surfaces of the upper slices of specimen 2 showed radial cracks in the central grout plug with no cracking in the anhydrite. The surfaces of the lower slices contained only map cracking on the central grout plug and no cracking in the anhydrite. This specimen was 93 days old when it was examined.

13. X-ray diffraction examination of grout from the different slices of specimens 2 and A7 revealed that tetracalcium aluminate dichloride-10-hydrate was restricted to the upper 1/4 in. of each specimen, showing that penetration by the brine during underwater storage was limited to this depth. The usual crystalline hydration products, ettringite and calcium hydroxide, were present in all the grout examined.

14. A sawed end surface of a length of anhydrite core was examined before a smaller core was removed and the rock was used to make specimen A7. Figure 2 shows that the rock was free of visible cracks before it

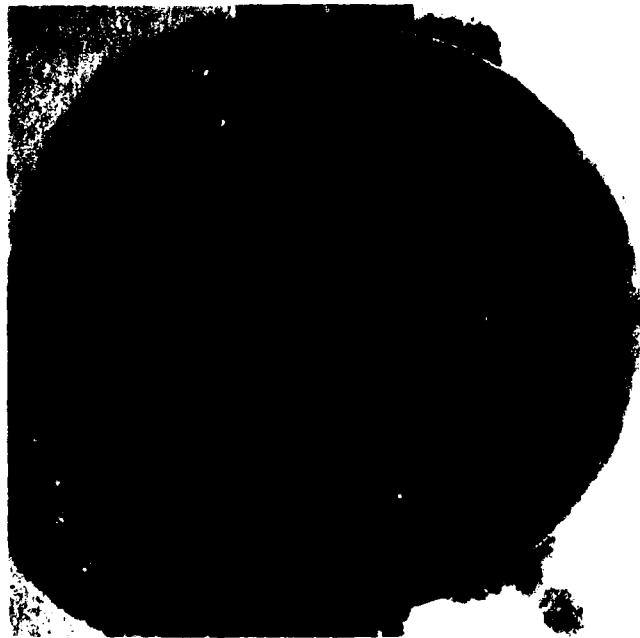


Figure 2. Wet sawed surface of anhydrite rock core used to make specimen A7, X0.8. Surface had no visible cracks

was used in a simulated borehole specimen.

15. A sawed surface of specimen A7 was examined while the steel restraining ring was still in place. No visible cracks were present in the rock or the grout. The steel restraining ring was removed, and significant cracking of the rock developed overnight (Figure 3a). Figure 3b shows a fracture surface from the same specimen with the steel restraining ring in place; the rock is not cracked and did not develop cracks. The grout in the approximate center of these two photographs appears cracked, but it is not; this appearance results from the grout being poured into water and displacing the water. It is believed that the white lines in the grout in Figure 3 represent thin layers of grout of high water content or concentrations of salt. This pair of photographs shows that the steel restraining ring is needed to prevent stress relief cracking when this type of specimen is being examined.

16. No cracking pattern was observed in specimen A10 when it was cut. An open crack developed at the interface between grout and anhydrite, with other cracks developing normal to this contact after the specimen was dried in a vacuum dessicator overnight at 140°F (60°C). Figure 4 shows the development of the cracking.

17. There was not usually enough of the white material that is shown at the contact surface (Figure 4a) to make a satisfactory specimen for examination by X-ray diffraction. When a portion of specimen 2 was cut, some of the contact between the rock and the outer layer of grout opened to reveal small amounts of a white secondary reaction product. X-ray diffraction examination of this material showed it to be composed of anhydrite, calcium hydroxide, and gypsum. The presence of gypsum suggests that there was some chemical reaction in the contact surface. The reaction was probably a solution of the anhydrous calcium sulfate (anhydrite) by water from the grout mixture and subsequent precipitation as hydrated calcium sulfate (gypsum).

18. The type of specimen that has been described in this work is intended to simulate the contact of grout to surrounding host rock, in this case anhydrite, from a formation plugged in a borehole. Assuming that the simulation is satisfactory, several points can be made:



a. Original tight contact of rock and grout

b. After overnight vacuum-drying at 140° F (60°C). Note that contact surface is open and grout is cracking radially

Figure 4. Sawed surface of specimen A10, X6 (anhydrite rock indicated by "A"; grout by "G")



a. Sawed surface with steel restraining ring removed. Note the cracks in the anhydrite rock and the eccentricity

b. Fracture surface with steel restraining ring in place. Note that the anhydrite rock is not cracked

Figure 3. Specimen A7, X0.7 (anhydrite rock indicated by "A")

- a. A fresh interior surface of the specimen needs to be exposed for examination of the contact area. A satisfactory way to obtain this surface is to cut through the metal pipe with a pipe cutter or a saw and then break through the specimen at that point to expose a fresh fracture surface.
- b. The procedure in (a) above seems preferable to sawing because sawing dry tends to smear the contact area and may generate enough heat to cause change. If sawing with a lubricant is used, water should be avoided since it can dissolve some anhydrite at the contact, while sawing with oil means that the oil will need to be removed before the new surface is examined.
- c. In any case, the steel restraining ring should be kept in place on slices of a specimen to prevent stress relief cracking of the rock and at the contact.
- d. The grout should not be dried since drying is likely to cause cracking at the contact or in the grout. Drying a specimen before examination with a scanning electron microscope (SEM) is usually necessary; this means that SEM examination should not be made unless special precautions are used to control cracking. This will be discussed in Part II.
- e. Examination by X-ray diffraction was made of grout specimens containing fresh mixing water. The specimens were placed and immediately stored under brine. The examination showed that penetration of the brine into the grout was limited to a depth of about 1/4 in. into the exposed grout surface at the time the specimens stored in brine were examined.
- f. X-ray diffraction results indicated that there was some solution of anhydrite at the contact with the grout and then precipitation as gypsum in the same area.

Preliminary Conclusions

19. Simulated borehole specimens of the type described in this report merit studying as a simulation of actual conditions in a borehole that is plugged with grout.

20. Based on the results obtained, the preferable way to study the surface where grout and rock are in contact is to use a stereomicroscope to examine a fresh fracture surface that has been kept wet and has its steel restraining ring still in place. Such examination will

determine whether there is cracking and whether the contact between the grout and rock surface is open or closed.

PART II: SECOND STUDY

Introduction

21. As was stated in Part I of this report, factors affecting the quality of the seal between the grout and the host rock are of prime importance. The contact between the sealing materials and the rock needs to be tight so that the interface will be effectively impermeable to liquids and to gases. The limited previous work described in Part I indicated that examination should be conducted using a stereomicroscope to observe features of wet fracture surfaces with the steel restraining ring in place.

22. The work described in this part was done to test this indication and to consider further the feasibility of examining features of these contact surfaces using a scanning electron microscope (SEM).

Experimental Procedure

23. Descriptions of simulated borehole (SBH) specimens intended to simulate downhole conditions have been given previously by Gulick et al. (1980a). Some of the SBH specimens used in this work were similar to those described by these researchers; others differed only by being smaller. The smaller specimens had overall dimensions of 2-3/4 in. in outside diameter and 3 in. in length. The smaller specimens were developed to be a more convenient size for handling during sample preparation for SEM examination. Six of these small SBH specimens were cast. Three were used for stereomicroscope examination; two were used for SEM examination. The five specimens that were examined by SEM are described in the tabulation below. The BCT-1-FF grout mixture was used in two field plugs (Gulick et al. 1980a). (This mixture is described in paragraphs 4 and 5.) Additional calcium sulfate hemihydrate was added to that in the mixture so that the mixing water was saturated with calcium sulfate.

Specimen No.	Mixing Water and Grout	Date Cast 1980	Age at Time of Examination	Specimen Preparation for SEM Examination
SBH-1a	Fresh water, BCT-1-FF	16 May	12 days	Freeze-dried, coated
SBH-1b*	Fresh water, BCT-1-FF	16 May	12 days	Freeze-dried, uncoated
SBH-2a	Fresh water, BCT-1-FF	16 May	12 days	Vacuum-dried at 140°F (60°C), coated
SBH-3a	CaSO ₄ -saturated BCT-1-FF	20 Jun	5 days	Freeze-dried, coated
SBH-4a	CaSO ₄ -saturated BCT-1-FF	20 Jun	12 days	Vacuum-dried at 140°F (60°C), coated

* This was a fractured mirror image of specimen SBH-1a.

24. Specimens were cast as follows:

- a. The length of steel pipe was placed on a glass plate.
- b. A piece of rock core with a hole in its center was placed on end and centered within the pipe.
- c. Freshly mixed grout was poured into the hole in the rock and into the space between the rock core and the pipe.
- d. Sometimes the space between the rock and the pipe was filled and allowed to harden. This was not the contact surface that was examined. The hole in the rock was filled at a later date.
- e. Once the hole in the rock was filled, the assembled SBH specimen was lowered into brine and kept there at 100°F (38°C) until it was examined.

Specimens SBH-1a, 1b, and 2a were the small size. They were filled in rapid sequence. Specimens SBH-3a and 4a were the large type. The outer annulus was filled with an expansive cement mixture and allowed to harden. The hole in the rock was later filled with the test grout mixture (Gulick et al. 1980b).

25. Fresh fracture surfaces were prepared by cutting through the metal pipe with a bandsaw. The specimen was then broken along the sawed

cut by applying pressure in three-point flexural loading. This satisfied the requirements for a fresh, wet fracture surface with metal restraining ring in place (Part I). The fracture surfaces were immediately examined with a stereomicroscope at magnifications ranging from 10 to 60X. This was done to three specimens to determine whether the host rock-to-grout contact had a good bond. The specimens to be viewed by the SEM were prepared by the freeze-dried or vacuum-dried methods before they were fractured.

26. The SBH specimens that were used for SEM examination were prepared as follows:

- a. Freeze-drying method. A Virtis manifold freeze-dryer was used. Dry ice (solid CO₂) was crushed and packed into the condenser. A vacuum pump was connected to the freeze-dryer and the system was evacuated. The specimen was placed in a 500-ml freeze-drying flask attached to the condenser and left for approximately 8 hours, or until the flask felt at room temperature.
- b. Vacuum dessicator method. A vacuum dessicator was placed in an oven at 140°F (60°C) and a mechanical vacuum pump was attached to the vacuum dessicator. The specimen was placed in the vacuum dessicator and the vacuum pump turned on. The specimen was left in the vacuum dessicator for approximately 16 hours.

After one of the drying procedures, the specimen was placed in a vacuum evaporator and subjected to further evacuation as needed before coating with a layer of carbon about 5 nm thick and a layer of gold-palladium alloy (80:20) about 15 nm thick to make the specimen electrically conductive. As indicated in the tabulation of specimens, one was not coated before SEM examination. It was examined at low voltage and magnification. The coated specimens were examined by SEM in the usual way. All of these specimens were again examined with a stereomicroscope before the SEM examination.

27. The examination of fresh fracture surfaces of SBH specimens with a stereomicroscope was improved by development of the following technique to keep the surface wet during examination:

- a. As soon as a specimen was broken, the fresh fracture surface was washed briefly in running tap water to remove loose particles and salt water.

- b. The specimen was then immersed in a container of distilled water with a fracture surface slightly below the surface of the water.
- c. The immersed surface was examined with a stereomicroscope without any drying taking place.

Results

28. The appearance of a fresh fracture surface of an SBH specimen is shown in Figure 5.

29. The contact between the grout and anhydrite was uniformly tight. There was no detectable cracking on any fresh fracture surface of SBH specimens examined before drying had taken place. Figure 6 shows the typical appearance of such a contact.

30. By contrast, these contacts always opened and cracking of the grout also usually occurred when drying was part of the specimen preparation procedure. Such cracks are shown in Figures 7 and 8. (Figure 8 is an SEM micrograph.)

31. There did appear to be some difference in the amount of cracking with the freshwater grout, according to the drying procedure, and freeze-drying seemed to cause more cracking. It had been thought the freeze-drying might be a preferable way to prepare these specimens for SEM examination, but such was not the case. Cracking of the SBH specimens filled with grout made with calcium sulfate saturated water seemed similar for both methods of drying.

32. The rinsing and immersion procedure described earlier for fresh fracture surfaces of SBH specimens prior to examination with a stereomicroscope proved to be quite satisfactory. No cracking was observed on specimen surfaces examined using this procedure.

Conclusions

33. Drying of SBH specimens, whatever the method of drying, was found in the earlier and later work to cause cracking of the specimen, especially along the contact surface between the grout and anhydrite. This



Figure 5. A typical SBH fracture surface shows a metal restraining ring on the outside perimeter next to the outer grout ring. Inside this grout is the anhydrite annulus filled with grout. This inside contact is the one that is examined. The SBH specimens range in size from that shown above, approximately 3 in. in diameter, to a larger size used for permeability testing, approximately 1 in. in diameter.

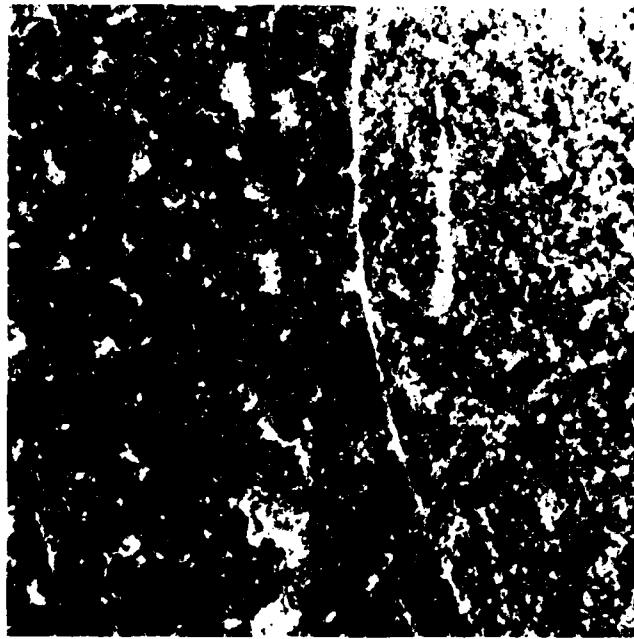


Figure 6. Appearance of a typical tight contact between grout (below) and anhydrite (above) on a fresh sample surface of an SBH specimen before any driving and associated cracking has occurred.

XII

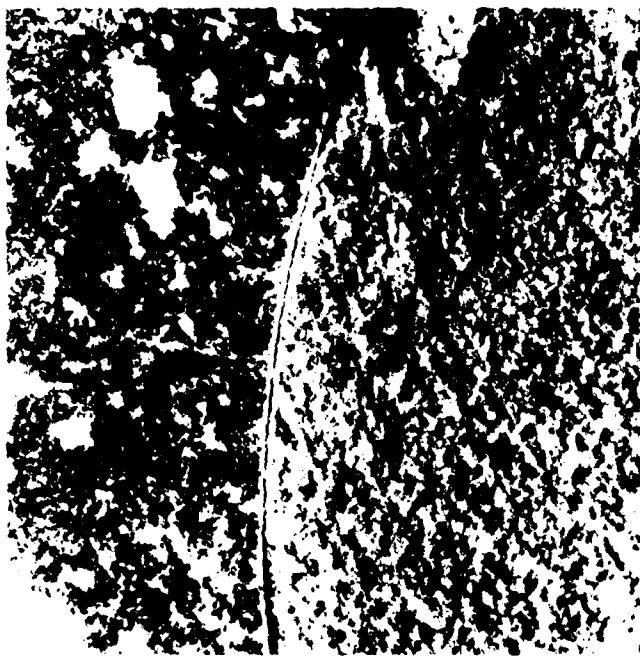


Figure 7. Typical cracking after drying, X13. The fractured surface was vacuum-dried at 140 °F (60 °C) for 16 hours



Figure 8. SEM micrograph 052980-5, X60. Note the crack along the contact between anhydrite (left) and grout (right), with another crack perpendicular to the opening running into the grout. This shows the damage done to a specimen during specimen preparation that involves drying. This is specimen S81-1a

may be expected to be true for other kinds of rock that might serve as host rocks. Therefore, it is recommended that drying not be a part of specimen preparation prior to examination or other testing involving this contact surface.

34. The procedure described herein of keeping a fresh SBH fracture surface continuously wet before and during examination with a stereomicroscope is recommended for examining grout-to-rock contact in such specimens.

35. The findings of this study agree with and extend the results of earlier work reported in Part I.

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EXAMINATIONS OF SIMULATED BOREHOLE SPECIMENS.(U)
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Errata Sheet

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1. Pages 10 and 11: The caption, including subtitles, for the photographs on page 10 should be under the photographs on page 11, and that for the photographs on page 11 should be under the photographs on page 10.